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(54) A PROCESS FOR THE MANUFACTURE OF DRAGÉES

(71) We, F. HOFFMAN-LA ROCHE & CO., AKTIENGESELLSCHAFT, a Swiss Company of 124-184 Grenzacherstrasse, Basle, Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for the manufacture of dragées, and is especially concerned with continuously measuring the temperature of the dragées during the coating process and controlling the process sequence accordingly.

In principle, the coating process consists in applying several layers of a coating mass to a dragée nucleus which, for example in the case of pharmaceutical dragées, usually contains the active substance. The coating mass can, for example, consist of a mixture of sugar, fillers and dyes. For the coating procedure, this coating mass is present in concentrated aqueous solution, called syrup, and is infused or sprayed in small portions onto the nuclei or dragées rotating in the coating drum. After each infusion, the syrup applied is uniformly distributed by the thorough mixing of the dragées constantly taking place in the coating drum, and the water contained in the syrup is subsequently removed by drying by introducing cold or warm air.

The quality of the finished dragées depends upon the degree of drying of the individual coating layers. If the moisture content of a layer is too high due to drying for too short a period of time, then the dragées will have poor storage characteristics and an irregular form. To long a drying time is uneconomical and for example, results in a strong dust-formation as well as a rough surface.

The degree of drying of the individual layers and thus the proper time of the respective next-following infusion can be determined subjectively by the coater using as a criteria the noise of rotation, rotary motion and dust-formation. These criteria cannot be employed in an automatic coating process.

Attempts have already been undertaken to determine objectively with the aid of other criteria the optimal degree of drying of the individual layers before the next-following infusion and, therewith, to automate the process.

Thus, attempts have been made to determine the moisture content of the dragées gravimetrically or titrimetrically. With this method, however, the measuring time is so great that the time for a further syrup infusion cannot be calculated quickly enough.

The method of determining the relative moisture hygrometrically via a small dragée sample has the same disadvantages.

Another attempt to determine when a coating layer is dry enough for the next coating operation was to hygrometrically measure the relative humidity in the ingoing and outgoing air thus obtaining a control parameter. However, in consequence of the large amounts of air and the therefore small humidity difference in the ingoing and outgoing air, this method is not accurate enough to obtain optimal values. Additionally, hygrometers are very sensitive to dust and therefore subject to disturbance in this procedure.

Another procedure consists in allowing the process to run according to a fixed time program. In so doing, however, good results can only be achieved when all influencing variables can be held absolutely constant. The disadvantage of this procedure accordingly consists in that the batch sizes, the aeration properties, the ambient temperature, the nucleus properties, the amount of outgoing air and, possibly, further variables have to be measured very accurately, which causes a very high technical expenditure. Small deviations from a required value cannot be determined with each individual infusion, but they accumulate since the control involves no automatic correction.

Accordingly, the process of the present invention aims at providing a control of the coating process which displays none of the disadvantages connected with the known processes. The temperature of the dragées is employed as the control parameter, since it

has been found that it is correlated with the degree of drying of the dragées.

The present invention therefore provides a dragee coating process which includes rotating the dragées in a coating drum and repeatedly applying and drying a certain amount of a coating mass thereon, comprising measuring the temperature of the dragées by means of a temperature sensor, converting the temperature measured into an electrical signal and releasing an application of coating mass whenever the signal reaches a predetermined value.

The temperature of the dragées falls after each infusion of syrup, due to the consumption of energy required for evaporation of the solvent. With falling moisture content, this energy consumption becomes smaller in the course of the drying process, and the dragée temperature again rises. When the temperature originally present before the infusion started is reached again, the total moisture applied is thus removed, and the folowing infusion can be undertaken. The criteria for the introduction of syrup must accordingly be derived from this point of the temperature curve.

The reference temperature which characterises the dry state can be chosen as desired and permits an adjustment of the desired residual moisture.

The temperature of the dragée nuclei or dragées is measured with a temperature sensor, for example a thermo-element. This temperature sensor can be fitted in a probe which dips into the rotating dragée nuclei or dragées in the coating drum.

The syrup infusion can be accomplished by any known means. Generally a ladle or spraying device is used. In order to insure uniform distribution of the syrup, the drying air can be switched off for a short time so as to delay the beginning of the drying. The course of the temperature can be registered with a pen recorder. There is thus obtained a fabrication record which gives information on the course of coating and the degree of drying.

For automatic systems, the temperature measuring apparatus is provided with a signal generator, for example, in the form of an end-contact, which at the point at which the reference temperature is again reached releases a control signal.

In one embodiment of the process in accordance with the invention, the control signal consists in an optical indication which gives the coater time for turning off the drying air and applying the next amount of syrup. With this embodiment, the advantage consists in that the time can be determined objectively and no longer lies in the judgement of the coater.

The process can also be carried out completely automatically, the control signal automatically (e.g. with the aid of known devices) bringing about the switching off of the drying air and the spraying on of an amount of syrup measured off with a dosage pump. At the same time, there can be switched on by the control signal a timer which, after an empirically determined interval which suffices for the uniform distribution of the syrup onto the total amount of dragées, again switches on the drying air.

The following Example illustrates the invention:

Example

A coating drum of 120 cm diameter is charged with 50 kg of dragée nuclei and set into rotation. The temperature of the dragée nuclei is continuously measured with a temperature sensor (Ni-CrNi thermoelement in a protective mantle of V2A steel) which is fixed to the suction hood over the coating drum and dips into the rotating dragée nucleus mass. The temperature is made visible on a display instrument which is equipped with an adjustable end-contact. The rotating dragée nuclei are blown with hot air of about 80°C and heated to 42°C. On reaching this temperature, a signal lamp is switched on by the end-contact which is adjusted to 42°C. On the basis of the signal, the ingoing air is turned off and the first infusion of 3 dl of white coating syrup is applied.

The time which is required for the uniform distribution of the syrup on the dragée nuclei amounts to 60 secs. The ingoing air of about 80°C is thereafter again switched on and the drying begins. The temperature of the dragée nucleus mass sinks to 39°C, extinguishing the signal lamp. In the course of about 4 minutes, the total moisture contained in the syrup evaporates and the temperature again rises to 42°C, actuating the signal lamp to give the time for the next infusion. In several successive layers the white coating and the colour coating is thus applied. After drying the last layer, the dragées are smoothed and polished in a known manner.

WHAT WE CLAIM IS:—

1. A dragée coating process which includes rotating the dragées in a coating drum and repeatedly applying and drying a certain amount of a coating mass thereon, comprising measuring the temperature of the dragées by means of a temperature sensor, converting the temperature measured into an electrical signal and releasing an application of coating mass whenever the signal reaches a predetermined value.

2. A dragée coating process acording to claim 1, wherein synchronously with the application of coating mass the drying is interrupted for a certain period of time

in order to allow better distribution of the coating mass.

3. A dragée coating process according to claim 1 or claim 2, wherein the electrical signal is recorded.

4. A dragée coating process according to claim 1, substantially as hereinbefore described with reference to the foregoing Example.

5. Dragées, when manufactured by the process claimed in any one of the preceding claims.

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